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	i		D OFFICE (DO/EO/US)	U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR		
	(CONCERNING A FILIN	G UNDER 35 U.S.C. 371	09/787496		
<u> </u>	NATI P	ONAL APPLICATION NO. CT/AU96/00791	INTERNATIONAL FILING DATE 17 September 1999	PRIORITY DATE CLAIMED 18 September 1998		
APPLIC Ceith David	CANT Mai	M HEATING ELEMENT (S) FOR DO/EO/US io TORPY GEHRIG				
Applic	ant h	erewith submits to the United Stat	es Designated/Elected Office (DO/EO/US)	the following items and other information:		
1.	\boxtimes		ems concerning a filing under 35 U.S.C. 3			
2.			UENT submission of items concerning a fi			
3.		This is an express request to begin examination until the expiration	n national examination procedures (35 U.S of the applicable time limit set in 35 U.S.C	S.C. 371(f)) at any time rather than delay 1. 371(b) and PCT Articles 22 and 39(1).		
4.				the 19th month from the earliest claimed priority date		
5.	\boxtimes	A copy of the International Appl	ication as filed (35 U.S.C. 371 (c) (2))			
		a. 🛭 is transmitted herewith	(required only if not transmitted by the In-	ternational Bureau).		
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			pplication was filed in the United States Re			
6.			Application into English (35 U.S.C. 371(c	c)(2)).		
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			by the International Bureau. Source, the time limit for making such ame	endments has NOT expired.		
		d. \(\square\) have not been made; no				
9.		· · · · · · · · · · · · · · · · ·	to the claims under PCT Article 19 (35 U.	S.C. 371(c)(3)).		
10.						
11.		An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)). A copy of the International Preliminary Examination Report (PCT/IPEA/409).				
12.			ne International Preliminary Examination R			
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13.			ement under 37 CFR 1.97 and 1.98.	nee with 27 CER 2.39 and 2.31 is included		
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17.		A substitute specification. A change of power of attorney a	nd/or address letter			
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THIN FILM HEATING ELEMENT

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BACKGROUND OF THE INVENTION

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This invention relates to heating elements of the kind including an electrically conductive metal oxide film on an electrically insulating substrate.

Such devices are known, and may for example consist of a thin film of tin oxide deposited on a glass substrate by means of pyrolitic deposition.

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If such thin film heating elements are to be used in electrical appliances such as cooktops, it is desirable that they be capable of operating at high temperatures, up to 650°C. In applications such as electric kettles where the heating element is small, the element must be capable of handling high power densities, of the order of 10-20 Watts cm⁻². Prior art devices have not proved satisfactory in these conditions. It has been found by the present applicants that tin oxide layers tend to become unstable with increasing temperature, due to the tendency for the oxide to change state. It has also been found that where fluorine is employed as an electron donor or conductivity carrier the properties of the film change irreversibly with increasing temperature,

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We have also found that the tin chloride solutions used in the prior art, for example in the spray pyrolysis process, are not stable in conditions of high humidity due to their hygroscopic properties, and this can lead to lack of uniformity in the oxide films produced.

apparently due to the fluorine tending to leave the film at temperatures above 400°C.

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US Patent No. 4,889,974 of Auding, et al. describes thin film elements intended for temperatures beyond 600°C, using oxide films doped at high levels with pairs of compensating foreign atoms. The metal oxide films are doped with, maximally, 10 mol % of each of the foreign atoms compensating each other in pairs, the quantity of said acceptor-forming elements and said donor-forming elements differing maximally

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by 10%. The Auding patent describes the use of indium, boron, aluminium or zinc as the acceptor-forming dopant, and antimony or fluorine as the donor-forming dopant.

However, these films using stannic chloride have been found to be difficult to deposit in humid atmospheres and have been found to be unstable in the power densities of approximately 20 Watts per cm² required for rapid rise-time applications.

To the applicants' knowledge the films described in the Auding patent have not seen commercial use and are known only from this document.

SUMMARY OF THE INVENTION

The present applicants have found that a metal oxide layer of satisfactory stability in high power density applications may be obtained by doping with at least one and preferably two rare earth elements. The rare earth dopants are preferably cerium and lanthanum. Preferably these two rare earths are present in substantially equal concentrations. The presence of the rare earth dopants in the thin film layer has been found by the present applicants to have the effect of stabilising the oxidation state of the metal.

We have also found that stability at high temperatures may be obtained by further doping with equal quantities of donor and acceptor elements, and by avoiding the use of fluorine as a dopant. The preferred donor and acceptor elements for this purpose are respectively antimony and zinc.

In one aspect, the invention resides in a thin film electrical heating element including a layer of an electrically conducting metal oxide on an electrically insulating substrate, said metal oxide layer being doped with at least one rare earth element.

30 Preferably the metal oxide is deposited on the substrate by pyrolysis of an organometallic base solution containing the at least one rare earth element.

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3 5 In a preferred form the metal oxide layer is tin oxide and contains two rare earth elements such as cerium and lanthanum. 10 This aspect of the invention provides a thin film heating element which is capable of withstanding power densities of up to 10-20 Watts cm⁻² and/or temperatures in excess of 600°C. 15 In another aspect, the invention resides in a method for the manufacture of a thin film heating element including the step of depositing a layer of metal oxide onto an electrically insulating substrate by pyrolysis of an organometallic base solution 20 containing at least one rare earth element. Preferably the base solution contains both cerium and lanthanum in concentrations up 25 to 5 mol %. 15 We have found that superior results can be obtained if the film is prepared by spray pyrolysis from a solution of monobutyl tin trichloride. The stability of this material in 30 high humidity enables consistent results to be obtained across varying atmospheric conditions, by reducing premature oxidation.

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BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is graph showing the power dissipation versus time relationship for a thin film heating element made according to the invention.

Fig. 2 shows the relationship between temperature and power at steady state for five elements having power ratings between 500 and 1330 watts.

DESCRIPTION OF PREFERRED EMBODIMENTS

While some benefit will be obtained from quite low concentrations of the rare earth dopant, minimal effects will be observed with concentrations in the pyrolysis solution

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of 0.01 mol %, preferred concentrations of each of the cerium and lanthanum are between approximately 1.25 mol % and approximately 3.75 mol %. Preliminary tests have shown that stability of the metal oxide layer is maximised when substantially equal concentrations of two rare earth elements, such as cerium and lanthanum, are used. Generally speaking the concentration of these rare earths will be chosen as that which contributes to film stability at the power densities for which the film is intended. Best results for films intended for operation at 20 Watts cm⁻² have been obtained using equal concentrations of approximately 2.5 mol %.

The film is preferably doped with substantially equal quantities of donor and acceptor elements, the preferred dopants being antimony and zinc. The concentrations of both antimony and zinc will be influenced by the resistivity which is required. We have found base solution concentrations for these materials in the region of 2.8 mol % to be suitable for heating element applications.

A useful characteristic of such films in their application as heating elements arises from the positive temperature coefficient resistance of the film. This enables elements to be produced which are self-regulating, in that they will initially operate at a higher wattage and, with increasing temperature, stabilise at the lower design wattage.

The substrate material will of course be chosen to suit the application. Suitable substrates include glass ceramics, silicon nitrides and other ceramic substrates as well as metallic substrates coated with high-temperature stable, electrically-insulating materials.

The preferred substrate temperatures for applying the base solution with dopants range from 500 to 750°C. Preferably, for application at 500°C, post annealing at approximately 600°C for at least one hour is carried out to assist in stabilising the film.

Films according to this invention were manufactured from a solution using the spray pyrolysis process. For this purpose, monobutyl tin trichloride was used as a base

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solution, with 2.8 mol % antimony chloride, 2.8 mol % zinc chloride, 2.5 mol % cerium and 2.5 mol % lanthanum.

These films were fabricated with effective resistances of 26 ohm, 30 ohm and 45 ohm to enable heaters of 2.2 kW, 1.8 kW and 1.2 kW respectively to be used, powered by a 240V mains supply voltage. The films were selectively deposited using high temperature masking inks which were removed by brushing after deposition of the film. The films deposited had a high degree of transparency. The resistive properties of the heating elements remained unchanged after 3500 cycles (40 minutes on and 20 minutes off) at 650°C.

As indicated above, the positive temperature coefficient of resistance of these elements enables a self-regulating characteristic to be obtained, with an initially high power dissipation which may be of advantage in achieving more rapid rise to operating temperature. Fig. 1 shows the typical behaviour of the elements, where power dissipation is plotted against time of operation. As will be observed, the dissipation of the element commences at a high level and decreases as the resistance of the element increases with temperature, until a steady state condition is achieved at the design power consumption. Upon temporary cooling of the element, for example through contact with a cooler body to be heated, power dissipation will temporarily increase, assisting in achieving rapid heating

increase, assisting in achieving rapid heating.

Fig. 2 shows the relationship between temperature and power at steady state for five elements having power ratings between 500 and 1330 watts.

Life tests have shown that the films are particularly stable on inert substrates like quartz 96% silica in temperatures up to 650°C with power densities in excess of 15.5W/cm². The films on lower grades of glass ceramics having alkali impurities such as lithium and sodium were stable to 500°C at extremely high power densities.

Sheet resistances varying from around 60 ohms to above 400 ohms have been fabricated by varying the number of spray passes. The thin film thickness could be

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varied between 2000 Angstrom Units to around 14000 Angstrom Units by varying the number of spray passes. The films were deposited on various substrates including glass ceramics, alumina, silica glass and silicon nitride.

As well as their suitability in high temperature and/or high rise time applications, films made in accordance with the invention may be used in low temperature applications, such as comfort heating, refrigerating defrost, and general heating. Heating elements of tubular shape manufactured using the above technology can be used in heat exchangers for flow applications, air-conditioning re-heaters, hair dryers, washing and drying appliances, and can also be used as radiating surfaces.

While particular embodiments of this invention have been described, it will be evident to those skilled in the art that the present invention may be embodied in other specific forms without departing from the essential characteristics thereof. The present embodiments and examples are therefore to be considered in all respects as illustrative and not restrictive, the scope of the invention being indicated by the appended claims rather than the foregoing description, and all changes which come within the meaning and range of equivalency of the claims are therefore intended to be embraced therein.

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CLAIMS

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 A thin film electrical heating element including a layer of an electrically conducting metal oxide on an electrically insulating substrate, said metal oxide layer being doped with at least one rare earth element.

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2. A thin film heating element according to claim 1 wherein said metal oxide layer includes at least two rare earth elements.

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10 3. A thin film heating element according to claim 2 wherein said two rare earth elements are present in said metal oxide layer in substantially equal concentrations.

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4. A heating element according to claim 2 or 3 wherein said at least two rare earth elements include both cerium and lanthanum.

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5. A heating element according to claim 1 wherein said metal oxide is tin oxide.

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6. A heating element according to claim 2 wherein said metal oxide layer further includes substantially equal quantities of donor and acceptor elements.

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7. A heating element according to claim 6 wherein said donor and acceptor elements are respectively antimony and zinc.

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A heating element according to claim 6 wherein said metal oxide layer is
 substantially free of fluorine.

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9. A heating element according to claim 1 wherein said heating element is stable at a power density of 20 watts cm⁻².

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10. A heating element according to claim 1 wherein said heating element is stable at a temperature of 650°C.

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10	11. A thin film heating element according to claim 1 wherein said meta deposited on said substrate by pyrolysis of an organometallic base solution said at least one rare earth element.	
15	12. A thin film heating element according to claim 11 wherein the or excarth element is present in said solution at a concentration up to 5 mol %.	ach rare
20	 13. A thin film heating element according to claim 12 wherein said at 1 10 rare earth element includes both cerium and lanthanum. 	east one
25	 14. A thin film heating element according to claim 13 wherein cerium lanthanum are each present in said solution in the range of approximately to approximately 3.75 mol %. 	
30	15. A thin film heating element according to claim 14 wherein the conceach of cerium and lanthanum in said solution is approximately 2.5 mol %	
35	16. A thin film heating element according to claim 11 wherein said sol 20 further includes substantially equal quantities of donor and acceptor eleme	
40	17. A thin film heating element according to claim 16 wherein each of and acceptor elements are respectively antimony and zinc and are each presolution at a concentration of approximately 2.8 mol %.	
45	18. A thin film heating element according to claim 11 or 13 wherein sa solution is monobutyl tin trichloride.	uid base
5 0	 A method for the manufacture of a thin film heating element included of depositing a layer of metal oxide onto an electrically insulating substrate pyrolysis of an organometallic base solution containing at least one rare experience. 	e by

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20. A method according to claim 19 wherein said solution contains at least two rare earth elements.

- 21. A method according to claim 20 wherein said two rare earth elements are present in said solution in substantially equal concentrations.
 - 22. A method according to claim 19 wherein said at least one rare earth element is present in said solution in the range of approximately 1.25 mol % to approximately 3.75 mol %.
 - 23. A method according to claim 20 wherein said at least two rare earth element includes both cerium and lanthanum.
- 24. A method according to claim 23 wherein said cerium and lanthanum are eachpresent in said solution in substantially equal concentrations.
 - 25. A method according to claim 19 wherein said base solution is monobutyl tin trichlorde.
- 26. A method according to claim 19 wherein said solution further includes chlorides of at least one donor and at least one acceptor element, said donor chlorides and acceptor chlorides being present in said solution in substantially equal concentrations.
- 25 27. A method according to claim 26 wherein said donor chloride is antimony chloride and said acceptor chloride is zinc chloride.
 - 28. A method according to claim 19 wherein said solution is substantially free of fluorine.

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WO 00/18189 A method according to claim 19 further including the step of annealing said 29. metal oxide layer on said substrate for at least one hour at a temperature higher than the substrate temperature used during said pyrolysis.





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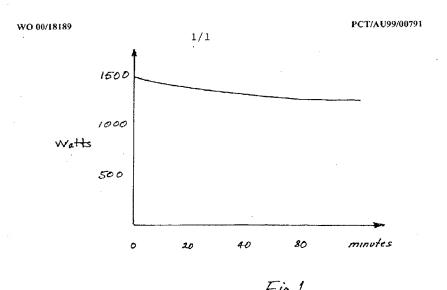
With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: SOLID ALUM SALT-BASED PRODUCTS IN A LIQUID DISPENSING CONTAINER

(57) Abstract: A method of creating solid alum salt-based products in a liquid dispensing container so that precise doses of alum salts may interface with water; forming useful commercial products with a far superior life span in the medicinal and personal care industries. Central to the invention is being able to form products that last up to 10 times longer than other products resulting in millions of tons less plastic wastage in the environment. Substantially this invention and art work create environmental based products because water is only to be added by consumers.



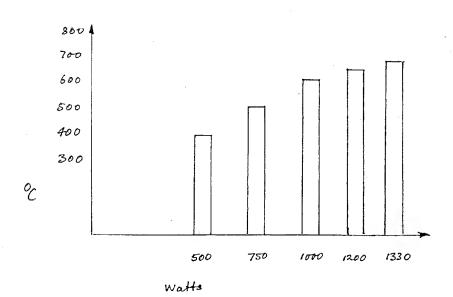


Fig. 2

Page 1 of

Docket No. 10032.00

Declaration and Power of Attorney For Patent Application

English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

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is attached here was filed on	eto. March 16, 2001	as United States Application No	, or PCT International
Application Nun	nber 09/787,496		
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-		nderstand the contents of the above amendment referred to above.	identified specification,
Section 1.56. nereby claim fore Section 365(b) of a PCT International sisted below and ha	eign priority benefits un ny foreign application(s application which design we also identified below to or PCT International	ility as defined in Title 37, Code of ander Title 35, United States Code, i) for patent or inventor's certificate, or gnated at least one country other tow, by checking the box, any foreign application having a filing date before	Section 119(a)-(d) or or Section 365(a) of any han the United States, application for patent or
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I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

PCT/AU99/00791	17 September 1999	Pending
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



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Full name of fourth inventor, if any	
Fourth inventor's signature	Date
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ill name of sixth inventor, if any	
xth inventor's signature	
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POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (list name and registration number)

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Beecroft 2119

Australia

ADDED PAGE TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR SIGNATURE BY JOINT INVENTOR(S) ON BEHALF OF NONSIGNING INVENTOR(S) WHO REFUSE(S) TO SIGN OR CANNOT BE REACHED (37 CFR 1.47(a))

WARRIMAG: "37 G.F.R. \$ 1.47(a) and 35 U.S.C. \$ 116 ¶ 2 require all available joint inventors to file an application 'on behalf' of themselves and on behalf of a joint inventor who 'carnot' be found or reached after diligent effort" "or who refuses to "join in an application." M.P.E.P. \$ 409.03(a), 6th ed., rev. 3 (amphesis edded). See also 62 Fed. Reg. 53,131, 53,137, 203 O.G. 68 (Oct. 10, 1997). I am an above named joint inventor and have signed this declaration on my own behalf and also sign this declaration under 37 CFR 1.47(a) on behalf of the nonsigning joint inventor, particulars for whom are: David M Gehrig Full name of (first, second, etc.) .. nonsigning inventor who refuses to sign ar reached cr reached NOTE: The name of the nonsigning inventor(s) should preferably also be filled in at the appropriate prior space. in the duclaration, adding the words "nonsigning inventor-completed on added page." Country of Citizenship of nonsigning inventor Australia Last known address of nonsigning inventor 39 Garfield Avenue, Bonnet Bay, NSW, 2226, NOTE: Ordinarily, the last known eddress will be the last known residence of the noneigning inventorial. A post office box is insufficient. Other addresses at which the nonsigning inventor(s) may be reached should iso be given. These can best be given in the Statement Of Facts in Support Of Filing On Behalf Of Omitted Inventor, MPEP § 409.03(e), 6th ad. II. Accompanying this declaration is: (1) A STATEMENT OF FACTS IN SUPPORT OF FILING ON BEHALF OF NONSIGN-ING INVENTOR (2) THE PETITION FEE OF \$130.00 (37 CFR 1.170) Keith Mario TORPY (type or print name of joint inventor signing on behalf of nonsigning Inventor) 22a Kirkham Street

(Added Page to Combined Declaration and Power of Attorney For Signature By One Joint Inventor on Behalf of Nonsigning Inventor(s) Who Refuse(s) to Sign or Cannot Be Reached (1-4)-page 1 of 1)